CDM

EPA Region 8

**Stimson Lumber Company
Libby, Montana**

December 2002

Draft Asbestos Sampling Report

Response Action Contract for Remedial, Enforcement Oversight, and Non-Time Critical Removal Activities at Sites of Release or Threatened Release of Hazardous Substances in EPA Region 8

Draft Asbestos Sampling Report
for
Stimson Lumber Company
Libby, Montana

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Acronyms

AHERA	Asbestos Hazard Emergency Response Act
ASTM	American Society for Testing and Materials
BZ	breathing zone
CDM	CDM Federal Programs Corporation
CFR	Code of Federal Regulations
CSS	contaminant screening study
EL	excursion limit
EPA	Environmental Protection Agency
EWS	extended work shift
f/cc	fibers per cubic centimeter
FJ	finger joint
ISO	International Organization for Standardization
LA	Libby Amphibole
lpm	liters per minute
MCE	mixed cellulose ester
MCS	MCS Environmental
mm	millimeter
ND	non-detect
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Safety and Health Administration
PCM	phase contrast microscopy
PEL	permissible exposure limit
PES	Pacific Environmental Services
RI	remedial investigation
SAP	sampling and analysis plan
SOP	standard operating procedure
Stimson	Stimson Lumber Company
TWA	time-weighted average
USDA	United States Department of Agriculture

cm^2	square centimeters
μm	micrometers

Section 1 Objective

The U. S. Environmental Protection Agency (EPA) has determined that materials originating from the Libby, Montana, vermiculite mine are sources of amphibole asbestos and should be removed (EPA 2002). Therefore, current investigations at residential and commercial properties in Libby focus on finding these source materials. However, because of the complexity of the Stimson Lumber Company (Stimson) facility relative to most properties under investigation in Libby, less was known about potential exposures, if any, that may result from disturbances of source materials. Therefore, the EPA determined that a more comprehensive investigation was required at Stimson than at other less complex commercial properties in Libby.

To investigate the potential impacts of these source materials, the EPA pursued a two-part approach. Initially, personal air, stationary air, and microvacuum dust samples were collected in areas where vermiculite is known or suspected to be present. These samples were collected to determine potential exposure information in these areas. The results of this air and dust sampling effort are discussed in this report. All sampling was conducted in accordance with the *Property Specific Sampling and Analysis Plan, Air and Dust Sampling, Stimson Lumber Company, Libby Asbestos Project, Libby, Montana,* (CDM Federal Programs Corporation [CDM] 2002a).

Secondly, the EPA conducted a study to screen all areas of the facility for potential amphibole asbestos sources, similar to the contaminant screening study (CSS) currently in progress at properties in Libby. This report will be updated to reflect the results of the study once analytical data is available.

The primary objective of this effort is to document and delineate potential sources of amphibole asbestos in a comprehensive, systematic manner.

The results of the two-part study are presented in this document. EPA will use this information to:

- Determine the need for any immediate actions
- Determine the need for removal actions
- Prioritize any future removal activities across the facility
- Determine the extent of contamination
- Delineate areas found not to contain amphibole asbestos contamination

1.1 Background

Historical information regarding the Stimson property in Libby, Montana, suggests that vermiculite products were used at, or transported to, the property at various times and at various locations. Much of this material is still present. Additionally, vermiculite insulation was installed in structures used for daily plant operations. It is believed that these products contain varying levels of the amphibole asbestos with compositions including tremolite, actinolite, richterite, and winchite (herein referred to as Libby Amphibole [LA]).

1.1.1 Site Location

Stimson is situated in the eastern section of Libby, Montana, on U. S. Highway 2 South (Figure 1-1). The facility is currently owned by Stimson Lumber Company and other private parties. The majority of the facility is currently used for manufacturing plywood board. The facility covers approximately 200 acres and encompasses processing, office, and other support buildings. In addition, a log yard and woodchip and mulch yard occupy a significant portion of the property.

1.1.2 Site History

The employee parking lot area used by Stimson employees was once used as an aboveground storage area for vermiculite insulation and is the location of the former popping plant facility. Vermiculite insulation was stockpiled directly on the native soil surface and may have contaminated the area with measurable amounts of asbestos mineral fibers. The area was converted to a parking lot in 1990.

A landscaping nursery was previously located along the southern boundary of the Stimson property. It is believed that unexfoliated, or raw vermiculite, was introduced to the site for use as a growth media and fill material. Currently the area remains a vacant lot with sparse vegetation. The lot is currently used to stockpile wood chips (collected from 1991 through 1997).

An overview of the facility layout is presented in Figures 1-2 and 1-3.

1.1.2.1 Site Visits

An initial site visit was conducted on September 28, 2001, by Dr. Chris Weis (EPA regional toxicologist), CDM, and Pacific Environmental Services (PES). Stimson personnel present during this meeting included Mr. Fred Sturgess (Libby complex manager), Ms. Veronica Bovee (health and safety coordinator), Mr. John Chopot (environmental manager), and Mr. Barry Brown (local union No. 2581 president). The site meeting included interviews with current employees and a walk-through of several areas of the facility.

A coordination meeting was held on September 9, 2002 at the Stimson site. Attendees included CDM and PES personnel. Stimson personnel present during this meeting included Ms. Veronica Bovee (Stimson health and safety coordinator), Mr. Fred Sturgess (Libby complex manager), and the operations managers for each building. Sampling locations and tasks were discussed, and a list of locations and personnel to be sampled was developed, as presented in the sampling and analysis plan (SAP). A

Color Map(s)

The following pages contain color that does not appear in the scanned images.

To view the actual images, please contact the Superfund Records Center at (303) 312-6473.

File Path: c:\projects\libby\libby_gis\aprs\stimson_sampling.apr

800 0 800 1600 Feet

Map Created November, 2002

N

CDM

LEGEND

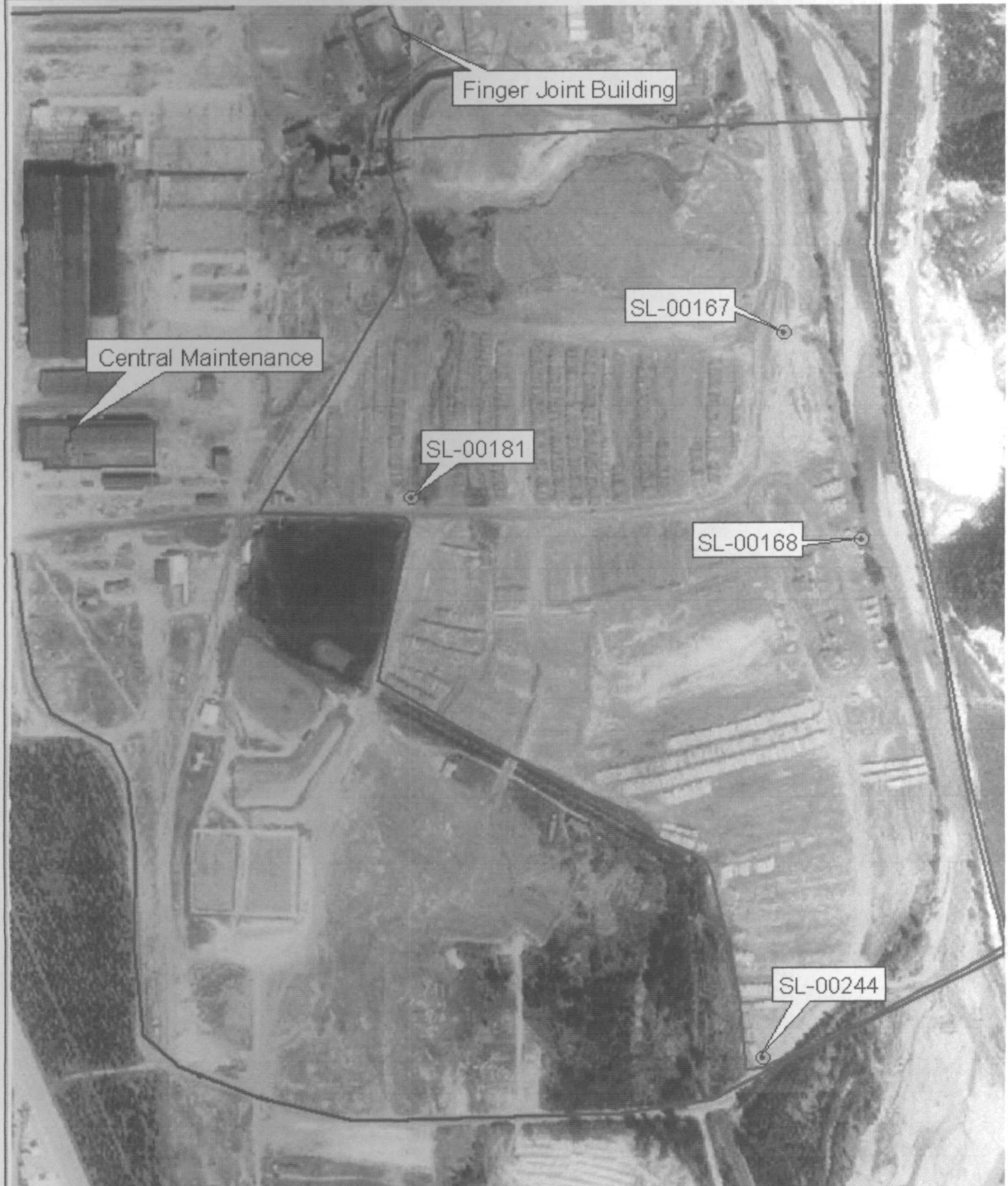
Stimson Lumber Property

Figure 1-1
Site Location Map
Stimson Lumber Company
Libby, MT

File Path: c:\projects\libby\libby_gis\aprs\stimson_sampling.apr

Figure 1-2
Stimson Lumber Company
Plywood Plant, Employee Parking Lot,
Finger Joint Building
Libby, MT

File Path: c:\projects\libby\libby_gis\aprs\stimson_sampling.apr



100 0 100 200 Feet

Map Created November, 2002

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Stimson Lumber Property

Areas of Concern

Outdoor Samples

Figure 1-3
Stimson Lumber Company
Central Maintenance, Logyard
Libby, MT

site visit was conducted to familiarize project personnel with the entire facility and the processes in each building to be sampled.

A progress meeting was held on September 17, 2002, at the Stimson office. Attendees included Mr. Greg Parana and Ms. Melissa Petrak of PES, and Ms. Veronica Bovee of Stimson. Topics discussed included:

- Samples collected to date, and plans for remaining sample collection
- Samples reported overloaded by the laboratory and resampling of the affected locations and tasks
- Buildings in which microvacuum dust samples would not be collected
- Bag house function and its affect on air samples collected in the plywood plant

An informal closeout meeting was scheduled for September 19, 2002, at the Stimson office. Ms. Veronica Bovee of Stimson was unavailable at the appointed time, so Ms. Melissa Petrak left a summary of samples collected for her records. Ms. Bovee was advised to contact the EPA Information Center with any questions or concerns.

1.1.2.2 Buildings Investigated as Part of Air and Dust Sampling

The central maintenance building currently contains vermiculite insulation. This structure is equipped with a large gantry crane that traverses the length of the building. As stated earlier, movement of this crane causes vibration within the structure and may release small amounts of vermiculite insulation from around seams and joints of the clapboard walls. There is a large main work area with vehicle bays, along with several smaller shops, a parts warehouse, locker room, break room and, supervisor's office. Central maintenance personnel work throughout the building on a variety of tasks, mostly involving vehicle and equipment maintenance and repair (Figure 1-3).

The plywood plant is currently used for processing plywood. Vermiculite insulation is believed to be associated with the big dryer No. 1 in this building. The plywood plant is one large open area, through which wood travels while being processed from logs to plywood. There are offices, break rooms, and rest rooms along the edges of the building. The debarker and log heating facility is outside and adjacent to the main plant structure (Figure 1-2). Plywood plant personnel generally work at their assigned task, although there is some rotation for relief purposes.

The finger joint (FJ) building is currently used for FJ operations. There is a main work area, along with the feeder No. 2 room, and the "wrap and stack" room. There is also a connection to Shed 12, which is a board storage area. FJ utility workers rotate tasks throughout the plant during the work shift, while a few employees work only on specific tasks. According to Veronica Bovee, the old lunchroom and bathroom area of the building previously contained vermiculite insulation, which was removed in May of 2000. The room is currently used for parts storage (Figure 1-2).

1.1.3 Environmental Setting

Mean annual precipitation in Libby is 19.4 inches, with 30 percent of it occurring in the months of November through January, and 18 percent falling in the months of May and June. The month having the highest average precipitation is January, with 2.42 in. Average ambient temperature in Libby ranges from 22.4°F in January, to 67°F in July. Average annual precipitation at the W.R. Grace vermiculite mine site is estimated at 20 inches per year (U. S. Department of Agriculture [USDA] 1977), and the temperature would be expected to average 3 to 5 degrees cooler than in Libby. Climatological data was obtained from the Libby 1 N.E. Ranger station.

1.1.4 Contaminant of Concern

The potential contaminant of potential concern investigated at this facility is asbestos. Asbestos fibers are odorless and tasteless and vary in length, structure, and chemical composition. Fibers are microscopic and environmentally persistent. They do not evaporate, burn or dry out from heat, or erode in water. Toxicity of different type fibers varies, but exposure to any one of them can be fatal. Tremolite, the form found at Libby, is considered by many to be the most toxic.

The human health risks associated with asbestos fibers released in the environment include:

- Asbestosis – a scarring of the lungs, which impairs elasticity of the lung
- Lung Cancer – a malignant tumor of the bronchi covering
- Mesothelioma – a cancer of the lining of the chest or of the abdominal wall
- Other diseases – increased incidence of some non-respiratory cancers has been seen in those exposed to asbestos

Asbestos related diseases have a latency period of 15 to 30 years, and the risks of asbestos exposure are significantly increased by smoking.

1.1.5 Previous Investigation

At the request of Stimson, MCS Environmental (MCS) performed industrial hygiene sampling to determine the potential exposure of Stimson employees to residual asbestos. Air samples taken within the central maintenance building and the plywood plant revealed concentrations of LA less than the Asbestos Hazard Emergency Response Act (AHERA) standard of <0.01 fibers/cubic centimeter (f/cc) for phase contrast microscope (PCM) analysis (40 Code of Federal Regulations [CFR] Part 763, 763.90 (i)(5).)

In addition, soil and bulk samples were taken from various locations around the facility including the central maintenance building, the former nursery, and the employee parking lot. While analysis of soils collected from the employee parking lots were all non-detect for asbestos, soils collected from the nursery area had concentrations of tremolite asbestos as high as 5 percent.

On May 2, 2002, two microvacuum dust samples were collected from the nursery shed. These samples, along with one field blank were analyzed by the International Standards Organization (ISO) 10312 method. The analysis of sample 1-06850 identified 5853 LA structures with lengths between 0.5 micrometers (μm) and 5 μm and 1170 LA structures with lengths between 5 μm and 10 μm. Sample 1-06850 was a composite of three locations in the nursery shed. No LA structures were detected on sample 1-06857, which was a composite of three locations on the floor of the nursery shed, or in sample 1-06858, the field blank. Analytical datasheets are included in Appendix A.

Section 2
Soil Sampling

Soil sampling at Stimson was designed for the quantification of relative LA abundance in soils throughout the site following all rationale, data quality objectives, quality assurance procedures, and standard operating procedures (SOPs) from the *Final Sampling and Analysis Plan (SAP) for the Remedial Investigation (RI) Contaminant Screening Study (CSS), Libby Asbestos Site, OU4* (CDM 2002b). For purposes of this investigation a site-specific SAP addendum was developed to the CSS SAP: *Final SAP Addendum for the Stimson Lumber Company Area, Libby Asbestos Site, OU4* (CDM 2002c). All soil investigation work was conducted in accordance with this SAP addendum.

To adequately characterize LA abundance in soils throughout Stimson, the site was divided into eight subareas (Figure 2-1): former popping plant, railroad spur, lumber yard, log storage yard, southwest area, former Champion International tree nursery, sprinkler field, and Champion International Superfund site. The Champion International Superfund site was appointed Superfund status due to groundwater contamination resulting from wood preservative processing and is not associated with LA contamination. Remediation efforts for the groundwater contamination are currently ongoing. These divisions were made based on assumed contaminant concentrations, land use, and environmental setting. During this investigation, no sampling was conducted within the sprinkler field or the Champion International Superfund site subareas as a result of ongoing remediation, and therefore, these subareas will not be discussed.

Surface and subsurface samples were collected from each subarea as follows:

Subarea	*Surface Soil Samples*	*Subsurface Soil Samples*
Former Popping Plant	16	7
Railroad Spur	14	0
Lumber Yard	25	6
Log Storage Yard	29	4
Southwest Area	14	3
Former Champion International Tree Nursery	11	8
Sprinkler Field	0	0
Champion Int'l Superfund Site	0	0
Total	**109**	**28**

Once results are available, this document will be updated.

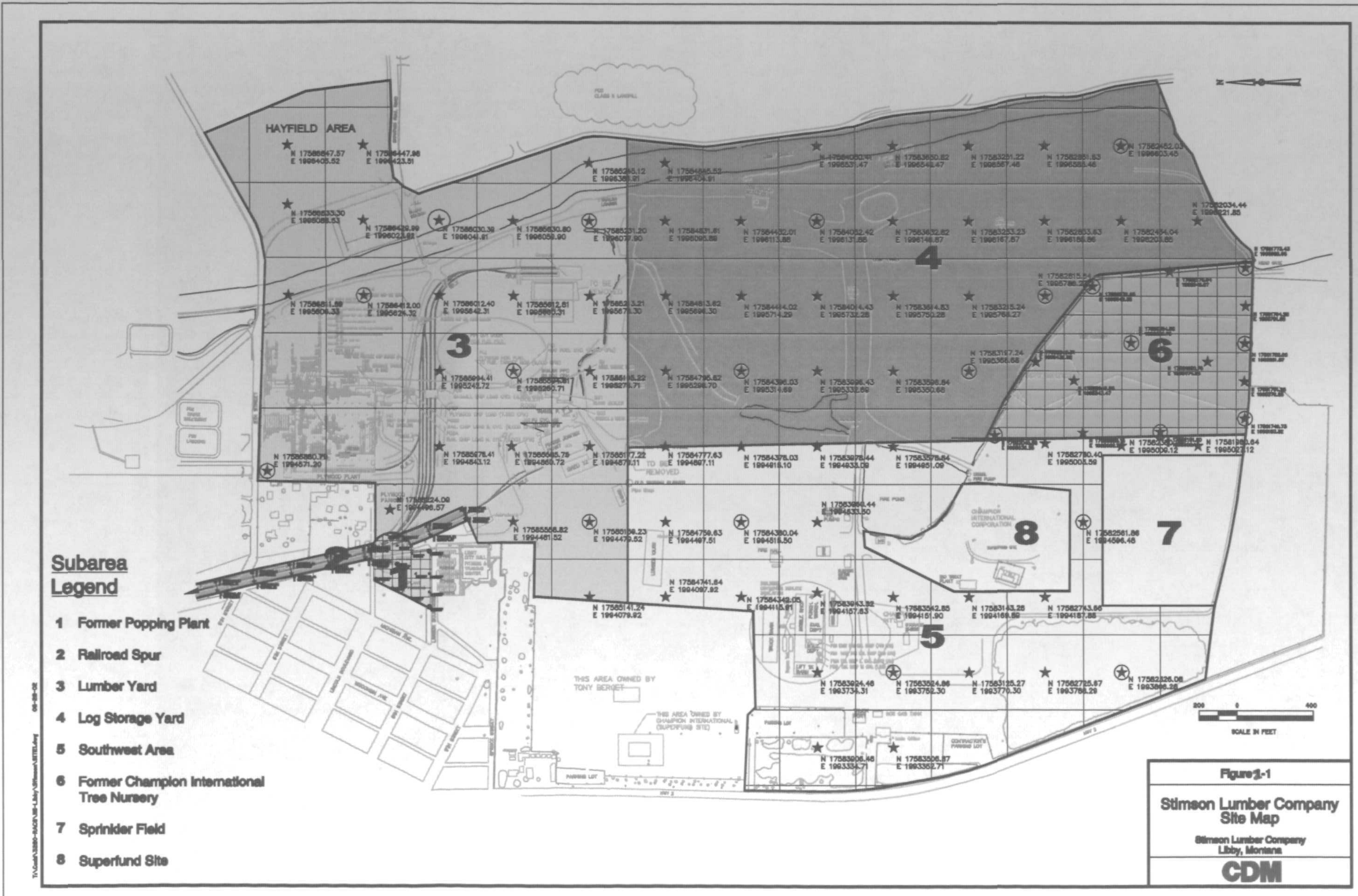

Figure 1-1

Stimson Lumber Company Site Map

Stimson Lumber Company
Libby, Montana

Section 3
Air Sampling

3.1 Personal Air Sampling

A total of 124 personal breathing zone (BZ) samples from 10 Stimson employees were collected. Ninety-seven air samples were collected for the duration of the work activity. The results of these samples were then calculated as time-weighted averages (TWAs) for the full shift (8, 10, or 12 hours) and compared to the Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) and/or the OSHA extended work shift PEL for asbestos. Twenty-seven samples were collected for 30 minutes (approximately) and compared to OSHA's 30-minute excursion limit (EL) for asbestos.

3.1.1 Sample Locations

Personal air sampling locations and tasks were selected during the pre-sampling facility visit on September 9, 2002. All locations and tasks were approved by EPA as presented in the SAP. These tasks represent normal and general duties typically performed by Stimson employees. Sampling locations and associated tasks at each location are summarized below:

- Plywood plant (Figure 1-2)
  - Dryer tender 1 – performed oversite on the dryers, troubleshooting, temperature, and steam tracking
  - Dryer feeder 2 – fed boards into dryer, general housekeeping
  - Dryer offbearer 3 – sorted and tended boards coming out of dryer
  - Plugger 4 – operated plugger machine
  - Green chain puller 5 – sorted wood from lathe along green chain
- Central maintenance building (Figure 1-3)
  - Mechanics (two employees) – performed repairs and maintenance on facility vehicles and machinery
- Finger joint building (Figure 1-2)
  - FJ utility – worked at all stations throughout FJ plant, including general housekeeping and forklift operation
- Log yard (Figure 1-3)
  - Wagner operator – operated Wagner Lumberjack, unloading and moving logs throughout log yard and to plywood plant area

Table 3-1 presents the number and type of samples collected at each task and location during the sampling activities.

Table 3-1
Task-Based Sample Numbers

Location	Task	No. of Samples Collected	
		Duration of Full Shift	30-Minute Excursion
Plywood Plant	Dryer tender	11	3
	Dryer feeder	12	3
	Dryer offbearer	11	3
	Plugger	9	3
	Green chain puller	13	3
Central Maintenance	Mechanic 1	6	3
	Mechanic 2	7	3
Finger joint	Finger joint utility	16	3
Log Yard	Wagner operator	12	3

3.1.2 Sample Collection

Personal air samples were collected on Stimson employees for 3 consecutive days, between September 10 and September 16, 2002. All samples were collected in accordance with the EPA Standard Operating Procedure (SOP) *2015 Asbestos Sampling* (Appendix B). Sample volume requirements were in accordance with OSHA *Construction Standard for Asbestos, 29 CFR 1926.1101*. All air sampling pumps were calibrated from 1.5 to 2.03 liters per minute (lpm) prior to the sampling period and again at the end of the sampling period. Air samples were collected using 0.8 μm open-faced 25 millimeter (mm) mixed cellulose ester (MCE) filters, as described in the SAP. All cassettes were visually inspected approximately every 2 hours during sampling to ensure cassettes were not overloaded.

Due to a higher level of airborne particulates than anticipated, cassettes were changed more frequently than every 2 hours in an effort to prevent sample overload.

For all samples collected during this investigation (personal, ambient, and microvacuum dust), field blanks were submitted at the rate of 10 percent and/or a minimum of two field blanks per sample batch per day. Field blanks were prepared at the time of sampling at the sampling location by removing the cassette cap for 30 seconds and then replacing the cap. A total of 26 field blanks were collected. All field blank cassettes originated from the field sampling cassette lot. One half of the field blanks submitted each day were analyzed, the remaining half were archived for later analysis if necessary. All field blanks analyzed during this investigation returned results of "ND," or no detected fibers at or above the detection limit. Field blank results are included in the Libby database printout presented in Appendix A.

Prior to receipt of new sampling cassettes in the field, a new unused cassette was sent to the laboratory. Results from these lot blanks determined the background asbestos structure concentration for the lot of cassettes. Specific cassette lot blanks analyzed during this project included 25mm three-piece cassettes with 0.8 μm MCE Filters for Lot

Numbers 21078 and 4583. All lot blanks analyzed during this investigation returned results of ND, or no detected fibers at or above the detection limit. Lot blank results are included in the Libby database printout presented in Appendix A.

Each employee selected to wear a pump was given a brief description of the purpose of personal sampling, as well as an explanation of the sampling procedure. This explanation included instruction to contact the sampling technician immediately if there were any problems with the sampling pump (e.g., pump falling off or stopping, or cassette loss or damage). This information was provided to each employee in writing and reviewed in person. Each of the selected employees provided their name, last 4 digits of their social security number (for sample tracking purposes), and job title on a form, which they signed and dated on the first day of sampling. A copy of this form is attached in Appendix C.

Employees wore pumps clipped to either the waistline of their pants, their own belt, or a belt that was provided. Tubing ran up their backs to the cassette, which was clipped to the collar or neckline of the employee's shirt, within the BZ, as described in the SAP. Clips or tape were sometimes used to secure tubing to the back of the employee's shirt, to prevent it from snagging or being caught on equipment. Employees were advised that the cassette must stay within their BZ for the duration of sample collection. Employees were questioned at the intervals when cassettes were visually inspected and at the end of each shift to determine if there were any problems with the sampling pump or cassette.

3.1.3 Sample Analysis

Air samples were analyzed by EMSL Analytical, Inc. (EMSL) in Libby, Montana, and Westmont, New Jersey; Reservoirs Environmental Services in Denver, Colorado, and/or Hygeia in Sierra Madre, California. All samples were analyzed in accordance with the ISO 10312, Air Quality - *Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method, 1995;* National Institute for Occupational Safety and Health (NIOSH) *Method 7400, Asbestos and other Fibers by Phase Contrast Microscopy (PCM);* and/or Appendix A of the EPA *Asbestos - Containing Materials in Schools: Final Rule and Notice*. If an air sample was determined to be overloaded, it was not analyzed by indirect preparation.

Samples were maintained under chain of custody procedures. After sample collection was completed, EPA custody labels were completed and affixed to each sampling cassette. Sample information was entered on chain of custody forms, and changes in sample custody noted on the form. Samples were relinquished to the sample coordinator and submitted for laboratory analysis. Any samples not immediately relinquished to the sample coordinator were secured in the sample storage cabinet. Samples were either hand-delivered to the EMSL onsite laboratory in Libby, Montana, or shipped via Federal Express to an offsite laboratory.

3.1.4 Summary of Results

The personal air samples were collected based on task. Each task was sampled for 3 consecutive days, for the duration of the work shift. One excursion limit sample was collected from each employee sampled on each of his or her 3 sampling days. A summary of TWA sample results, including calculated extended work shift values, is presented in Table 3-2. Extended work shift permissible exposure limits were determined using a standard OSHA formula (American Industrial Hygiene Journal 2000). A summary of excursion limit sample results is presented in Table 3-3. A complete Libby database printout of personal air sample results is attached in Appendix A.

Personal air samples were collected on two employees in the Central Maintenance building: Mechanic 1 and Mechanic 2. Both central maintenance employees worked an 8-hour shift each sampling day. Asbestos structures were detected by TEM AHERA analysis on one of the six samples (SL-00018) collected on Mechanic 1. Asbestos structures were detected by TEM AHERA analysis on two of the seven samples (SL-00012 and SL-00054) collected on Mechanic 2. TWA calculation based on the PCM analysis results showed no exposures above the OSHA PEL.

Personal air samples were collected on one employee in the FJ building: FJ Utility. FJ Utility worked a 10-hour shift each sampling day. Asbestos structures were detected by TEM AHERA analysis on one of the 16 samples collected on FJ Utility, SL-00051. One sample, SL-00198, was overloaded for PCM analysis, and therefore TWA calculation was not possible for that date. TWA calculation based on the PCM analysis results for the remaining dates showed no exposures above the PEL.

Personal air samples were collected on one employee in the Log yard: Wagner Operator. The Wagner operator worked an 8-hour shift each sampling day. Asbestos structures were detected by TEM AHERA analysis on one of the twelve samples collected on the Wagner Operator, SL-00055. Two samples, SL- 00166 and SL-00189, were overloaded for PCM analysis, and therefore TWA calculation was not possible for that date. TWA calculation based on the PCM analysis results for the remaining dates showed no exposures above the PEL. Extended work shift (EWS) TWA calculation based on the PCM analysis results for the remaining dates showed no exposures above the calculated EWA PEL.

Five employees were sampled in the Plywood Plant: Dryer Feeder, Dryer Tender, Dryer Offbearer, Green Chain Puller, and Plugger. The Dryer Feeder, Dryer Tender, and Dryer Offbearer worked 12-hour shifts the first 2 sampling days, and a 6-hour shift the 3rd day. The Green Chain Puller worked 10-hour shifts each of the 3 sampling days, and the Plugger worked 8-hour shifts each of the 3 days.

Asbestos structures were not detected by TEM AHERA analysis on any of the 11 samples collected on the Dryer Feeder. TWA calculation based on the PCM analysis

Table 3-2
Personal Air Sampling - TWA Extended Work Shift (EWS) Results

				PCM	TEM AHERA					
Building	**Task**	**Sample Date**	**Index ID**	**Lab Result (f/cc)**	**Lab Result (S/cc) ***	**Sample Time (min)**	**Work Shift (Hrs)**	**TWA ** 8-Hr (f/cc)**	**TWA-EWS (f/cc)**	**PEL-EWS (f/cc)**
Central Maintenance	Mechanic 1	10/Sep/02	SL-00002	< 0.005	ND	291	8	< 0.006	NA	NA
			SL-00011	< 0.008	ND	164				
Central Maintenance	Mechanic 1	11/Sep/02	SL-00018	< 0.005	0.005	291	8	< 0.006	NA	NA
			SL-00031	< 0.008	ND	166				
Central Maintenance	Mechanic 1	12/Sep/02	SL-00041	0.008	ND	173	8	< 0.006	NA	NA
			SL-00053	< 0.009	ND	146				
Central Maintenance	Mechanic 2	10/Sep/02	SL-00003	0.009	ND	292	8	< 0.008	NA	NA
			SL-00012	< 0.008	0.009	167				
Central Maintenance	Mechanic 2	11/Sep/02	SL-00019	< 0.004	ND	302	8	< 0.044	NA	NA
			SL-00032	< 0.119	ND	167				
Central Maintenance	Mechanic 2	12/Sep/02	SL-00042	0.008	ND	174	8	< 0.011	NA	NA
			SL-00048	0.021	ND	123				
			SL-00054	< 0.009	0.013	143				
Finger Joint	FJ Utility	10/Sep/02	SL-00001	0.03	ND	293	10	< 0.028	< 0.026	0.08
			SL-00009	0.02	ND	158				
			SL-00014	< 0.012	ND	115				
Finger Joint	FJ Utility	11/Sep/02	SL-00017	< 0.135	ND	147	10	< 0.086	< 0.055	0.08
			SL-00029	< 0.161	ND	123				
			SL-00035	0.015	ND	115				
Finger Joint	FJ Utility	12/Sep/02	SL-00040	0.035	ND	144	10	< 0.030	< 0.029	0.08
			SL-00045	< 0.013	ND	100				
			SL-00047	0.039	ND	55				
			SL-00051	0.023	0.013	117				
			SL-00057	0.017	ND	97				
			SL-00063	< 0.02	ND	69				
Finger Joint	FJ Utility	16/Sep/02	SL-00161	0.187	ND	157	10	overloaded	overloaded	0.08
			SL-00183	0.279	ND	126				
			SL-00198	overloaded	ND	173				
			SL-00206	0.059	ND	116				
Log Yard	Wagner Operator	10/Sep/02	SL-00005	< 0.088	ND	204	8	< 0.043	NA	NA
			SL-00010	< 0.01	ND	129				
			SL-00013	< 0.009	ND	143				
Log Yard	Wagner Operator	11/Sep/02	SL-00027	< 0.058	ND	172	8	< 0.026	NA	NA
			SL-00030	< 0.007	ND	198				
			SL-00033	< 0.012	ND	111				
Log Yard	Wagner Operator	12/Sep/02	SL-00044	< 0.141	ND	127	8	< 0.081	NA	NA
			SL-00050	< 0.163	ND	110				
			SL-00055	0.018	0.015	96				
			SL-00058	< 0.012	ND	109				
Log Yard	Wagner Operator	16/Sep/02	SL-00166	overloaded	ND	121	8	overloaded	NA	NA
			SL-00189	overloaded	ND	143				

Table 3-2
Personal Air Sampling - TWA Extended Work Shift (EWS) Results

Building	Task	Sample Date	Index ID	PCM Lab Result (f/cc)	TEM AHERA Lab Result (S/cc) *	Sample Time (min)	Work Shift (Hrs)	TWA ** 8-Hr (f/cc)	TWA-EWS (f/cc)	PEL-EWS (f/cc)
Plywood Plant	Dryer Offbearer	13/Sep/02	SL-00076	0.031	ND	188	12	< 0.037	< 0.032	0.07
			SL-00086	0.05	ND	139				
			SL-00104	< 0.01	ND	180				
			SL-00116	0.025	ND	118				
Plywood Plant	Dryer Offbearer	14/Sep/02	SL-00125	< 0.014	ND	124	12	< 0.094	< 0.086	0.07
			SL-00137	< 0.014	ND	128				
			SL-00146	0.084	ND	90				
			SL-00147	0.106	ND	126				
			SL-00153	0.109	ND	189				
Plywood Plant	Dryer Offbearer	16/Sep/02	SL-00160	0.05	ND	189	12	< 0.036	0.017	0.07
			SL-00187	0.048	ND	162				
Plywood Plant	Plugger	13/Sep/02	SL-00078	0.04	0.015	132	8	0.035	NA	NA
			SL-00088	0.039	ND	93				
			SL-00099	0.038	ND	204				
Plywood Plant	Plugger	14/Sep/02	SL-00131	< 0.015	ND	121	8	< 0.039	NA	NA
			SL-00141	< 0.015	ND	119				
			SL-00143	0.077	ND	195				
Plywood Plant	Plugger	16/Sep/02	SL-00165	0.048	ND	143	8	0.053	NA	NA
			SL-00188	0.113	ND	125				
			SL-00199	0.027	ND	169				
Plywood Plant	Dryer Feeder	13/Sep/02	SL-00074	0.034	ND	191	12	0.041	0.037	0.07
			SL-00085	0.029	ND	150				
			SL-00105	0.043	ND	39				
			SL-00101	0.024	ND	147				
			SL-00115	0.03	ND	118				
Plywood Plant	Dryer Feeder	14/Sep/02	SL-00124	< 0.015	ND	120	12	< 0.060	< 0.055	0.07
			SL-00134	< 0.014	ND	127				
			SL-00144	0.066	ND	119				
			SL-00150	0.035	ND	94				
			SL-00152	0.072	ND	197				
Plywood Plant	Dryer Feeder	16/Sep/02	SL-00157	0.026	ND	190	12	0.016	0.008	0.07
			SL-00185	0.018	ND	159				
Plywood Plant	Green Chain Puller	13/Sep/02	SL-00075	0.05	ND	179	10	0.056	0.054	0.08
			SL-00083	0.032	ND	166				
			SL-00093	0.036	ND	154				
			SL-00114	0.087	ND	79				
Plywood Plant	Green Chain Puller	14/Sep/02	SL-00123	< 0.015	ND	119	10	< 0.128	< 0.129	0.08
			SL-00133	< 0.014	ND	127				
			SL-00142	0.062	ND	114				
			SL-00148	0.128	ND	99				
			SL-00151	0.255	ND	149				

Table 3-2
Personal Air Sampling - TWA Extended Work Shift (EWS) Results

				PCM	TEM AHERA					
Building	Task	Sample Date	Index ID	Lab Result (f/cc)	Lab Result (S/cc) *	Sample Time (min)	Work Shift (Hrs)	TWA ** 8-Hr (f/cc)	TWA-EWS (f/cc)	PEL-EWS (f/cc)
Plywood Plant	Green Chain Puller	16/Sep/02	SL-00158	0.059	ND	190	10	0.054	0.055	0.08
			SL-00184	0.037	ND	176				
			SL-00200	0.026	ND	111				
			SL-00207	0.039	ND	134				
Plywood Plant	Dryer Tender	13/Sep/02	SL-00073	0.015	ND	196	12	0.032	0.031	0.07
			SL-00084	0.016	ND	269				
			SL-00110	0.044	ND	120				
			SL-00117	0.026	ND	113				
Plywood Plant	Dryer Tender	14/Sep/02	SL-00126	< 0.014	ND	124	12	< 0.093	< 0.087	0.07
			SL-00138	< 0.015	ND	117				
			SL-00145	0.095	ND	113				
			SL-00149	0.054	ND	135				
			SL-00154	0.125	ND	185				
Plywood Plant	Dryer Tender	16/Sep/02	SL-00159	0.024	0.014	187	12	0.022	0.011	0.07
			SL-00186	0.039	ND	160				

* ND - Indicates no Libby Amphibole structures detected by TEM AHERA analysis.
** TWA measured against PEL of 0.1 f/cc, accordance with OSHA 1926.1101.

NA - Indicates no extended work shift (EWS) PEL is required.
AHERA - Asbestos Hazardous Emergency Response Act
EWS - Extended work shift
f/cc - Fibers per cubic centimeter
Hr - Hour
min - Minutes
PCM - Phase contrast microscopy
S/cc - Structures per cubic centimeter
TEM - Transmission electron microscopy

Table 3-3
Excursion Air Sampling Results

					PCM	TEM AHERA
Building	***Task***	***Sample Date***	***Index ID***	***Sample Time (min)***	***Lab Result * (f/cc)***	***Lab Result ** (S/cc)***
Central Maintenance	Mechanic 1	9/10/2002	SL-00008	31	<0.043	ND
Central Maintenance	Mechanic 1	9/11/2002	SL-00025	30	<0.044	ND
Central Maintenance	Mechanic 1	9/12/2002	SL-00043	32	<0.041	ND
Central Maintenance	Mechanic 2	9/10/2002	SL-00007	30	<0.044	ND
Central Maintenance	Mechanic 2	9/11/2002	SL-00026	30	<0.044	0.049
Central Maintenance	Mechanic 2	9/12/2002	SL-00056	35	<0.038	ND
Finger Joint	FJ Utility	9/10/2002	SL-00004	34	0.064	ND
Finger Joint	FJ Utility	9/11/2002	SL-00028	31	<0.043	ND
Finger Joint	FJ Utility	9/12/2002	SL-00046	30	<0.044	ND
Log Yard	Wagner Operator	9/10/2002	SL-00006	32	<0.041	ND
Log Yard	Wagner Operator	9/11/2002	SL-00034	35	<0.038	ND
Log Yard	Wagner Operator	9/12/2002	SL-00052	30	<0.044	0.049
Plywood Plant	Dryer Feeder	9/13/2002	SL-00103	31	0.078	ND
Plywood Plant	Dryer Feeder	9/14/2002	SL-00135	30	<0.044	ND
Plywood Plant	Dryer Feeder	9/16/2002	SL-00191	31	<0.043	ND
Plywood Plant	Dryer Offbearer	9/13/2002	SL-00113	30	<0.044	ND
Plywood Plant	Dryer Offbearer	9/14/2002	SL-00136	30	<0.044	ND
Plywood Plant	Dryer Offbearer	9/16/2002	SL-00194	33	<0.04	ND
Plywood Plant	Dryer Tender	9/13/2002	SL-00109	30	<0.044	ND
Plywood Plant	Dryer Tender	9/14/2002	SL-00139	32	<0.041	ND
Plywood Plant	Dryer Tender	9/16/2002	SL-00193	31	<0.043	ND
Plywood Plant	Green Chain Puller	9/13/2002	SL-00100	30	<0.044	ND
Plywood Plant	Green Chain Puller	9/14/2002	SL-00132	30	<0.044	ND
Plywood Plant	Green Chain Puller	9/16/2002	SL-00192	30	<0.044	ND
Plywood Plant	Plugger	9/13/2002	SL-00095	32	<0.041	ND
Plywood Plant	Plugger	9/14/2002	SL-00140	36	<0.037	ND
Plywood Plant	Plugger	9/16/2002	SL-00190	30	<0.044	ND

* Result measured against Excursion Limit of 1.0 f/cc, in accordance with OSHA 1926.1101.

** ND indicates no Libby Amphibole structures detected by TEM AHERA analysis.

AHERA - Asbestos Hazardous Emergency Response Act
f/cc - Fibers per cubic centimeter
min - Minutes
PCM - Phase contrast microscopy
S/cc - Structures per cubic centimeter
TEM - Transmission electron microscopy

results showed no exposures above the PEL. EWS TWA calculation based on the PCM analysis results showed no exposures above the calculated EWS PEL.

Of the 11 samples collected on the Dryer Tender, asbestos structures were detected by TEM AHERA analysis on one sample, SL-00159. The TWA calculation based on the PCM analysis results for the remaining dates showed one exposure above the PEL. The calculated extended work shift TWA for September 14, 2002, was <0.087 f/cc, which is above the calculated extended work shift PEL of 0.07 f/cc. However, this overexposure is not conclusive, because the high limit of detection does not allow for a valid comparison between the values.

Asbestos structures were not detected by TEM AHERA analysis on any of the 11 samples collected on the Dryer Offbearer. TWA calculation based on the PCM analysis results showed no exposures above the PEL. EWS TWA calculation based on the PCM analysis results showed no exposures above the calculated EWS PEL.

Asbestos structures were not detected by TEM AHERA analysis on any of the 13 samples collected on the Green Chain Puller. TWA calculation based on the PCM analysis results for the remaining dates showed one exposure potentially above the PEL. The EWS TWA calculated for September 16, 2002, was <0.054 fibers per cubic centimeter (f/cc), which is less than the calculated EWS PEL of 0.08 f/cc.

Asbestos fibers were detected by TEM AHERA analysis on one of the nine samples collected on the Plugger, SL-00078. TWA calculation based on the PCM analysis results for the remaining dates showed no exposures above the PEL. ISO 10312 results for all personal samples are presented in Appendix A.

All excursion limit samples collected on Stimson employees showed PCM results significantly lower than the OSHA-defined excursion limit of 1.0 f/cc. Asbestos structures were detected on two excursion limit samples by TEM AHERA analysis: Samples SL-00026 and SL-00052. SL-00026 was collected on Mechanic 2 in central maintenance on September 11, 2002. SL-00052 was collected on the Wagner operator on September 12, 2002. Asbestos structures were not detected on the remaining excursion limit samples by TEM AHERA analysis. A summary of excursion limit sample results is presented in Table 3-3.

3.2 Ambient Air Sampling

A total of 43 ambient air samples were collected. These samples were collected inside buildings and outdoors to determine general background asbestos concentration levels at the Stimson facility. All locations were approved by EPA prior to sampling.

3.2.1 Sample Locations

As presented in the SAP, ambient air sampling was conducted in three facility buildings on the Stimson property. These buildings included the plywood plant, central maintenance building, and the FJ building (Figures 1-1 and 1-2). Ambient air sampling was also conducted at two outdoor locations on the Stimson property and included the employee parking lot (near the former popping plant) (Figure 1-2) and the log yard (Figure 1-3). Samples were collected during normal daily operations while facility equipment was operational. Sampling locations are summarized in Table 3-4.

Table 3-4
Ambient Air Sampling Locations

Location	*Sample Location*	*No. of Samples*
Central Maintenance (BD-002098)	Center of machine shop	1
	Center of south end of building	1
	East side center of building	1
	North end center of building	3
	Replicate – north end center of building	3
Plywood Plant (BD-002099)	Outside de-barker cab	1
	Green chain exterior wall opposite supervisor's office	4
	Plugger alley next to plugger No. 9	4
	Dryers next to post at feed end	4
	Spreaders at post near pre-press	3
	Inside de-barker cab	1
Finger Joint building (BD-002097)	Outside lunch room in main plant area	2
	Near entrance to feeder No. 2 room	2
	Near former lunch room	2
Employee Parking Lot	Southeast corner	1
	Northwest corner	1
	Center of south side of lot	1
	In railroad tracks, north of roadway	1
Log Yard	Outside log truck scale shed	2
	Outside storage shed	1
	At trailer crane by fire pond	2
	Near head gate	2

3.2.2 Sample Collection

All ambient air samples collected between September 11 and September 18, 2002, were collected according to the EPA Standard, *Operating Procedure (SOP) 2015 Asbestos Sampling* (Appendix B). All ambient air sampling pumps were calibrated prior to the sampling period and again at the end of the sampling period. Air samples were collected using 0.8 μm open-faced 25mm MCE filters. All air sampling cassettes were inspected during sampling to determine if file overloading was occurring.

All field blanks analyzed during this investigation returned results of ND, or no fibers detected at or above the detection limit. Field blank results are included in the Libby database printout presented in Appendix A.

Specific cassette lot blanks analyzed during this project included MCE filters for lot numbers 21078 and 4583. All lot blanks analyzed during this investigation returned results of ND, or no fibers detected at or above the detection limit for the analytical method. Lot blank results are included in the Libby database printout presented in Appendix A. As a means of assessing sample variability during ambient air sampling, three replicate samples from the central maintenance building were collected.

3.2.3 Sample Analysis

Air samples were analyzed by EMSL Analytical, Inc. in Libby, Montana, and Westmont, New Jersey; Reservoirs Environmental Services in Denver, Colorado, and/or Hygeia in Sierra Madre, California. All samples were analyzed by ISO 10312, Air Quality - *Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method, 1995;* NIOSH *Method 7400, Asbestos and other Fibers by Phase Contrast Microscopy (PCM);* and/or Appendix A of EPA *Asbestos - Containing Materials in Schools: Final Rule and Notice.*

Samples were maintained under chain of custody procedures. After sample collection was completed, EPA custody labels were completed and affixed to each sampling cassette. Sample information was entered on chain of custody forms, and changes in sample custody noted on the form. Samples were relinquished to the sample coordinator and submitted for laboratory analysis. Any samples not immediately relinquished to the sample coordinator were maintained under chain of custody. Samples were either hand-delivered to the EMSL onsite laboratory in Libby, Montana, or shipped via Federal Express to an offsite laboratory.

3.2.4 Summary of Results

A total of 42 stationary air samples were collected and analyzed in Stimson buildings. A summary of stationary sample locations and results, including PCM and TEM AHERA analysis results, is presented in Table 3-5. A complete list of results, including those for ISO 10312 analysis, is presented in Appendix A.

Of the nine samples collected in the Central Maintenance building, fibers were not detected at levels at or above 0.01 f/cc by PCM analysis. Asbestos structures were detected on one of the nine samples (SL-00223) by TEM AHERA analysis, collected at the center of the north end of the building.

Of the 15 samples collected throughout the Plywood Plant, fibers were detected at values at greater than 0.01 f/cc by PCM analysis in 7 samples. Three of the samples, SL-00079, SL-00092, and SL-00107, were located along the green chain exterior wall, opposite the supervisor's office. Two samples, SL-00243 and SL-00245, were located at the spreaders, at a post near the pre-press. The remaining three samples at or above 0.01 f/cc were SL – 00092, SL – 00106, and SL-00215. SL-00092 was located at plugger alley, next to plugger No. 9; SL-00106 was located at the dryers, at a post near the feed end; and SL-00215 was located in the debarker cab. Asbestos structures were not detected in any of the 15 samples by TEM AHERA analysis.

On five of the six samples collected in the FJ building, fibers were not detected at levels at or above 0.01 f/cc by PCM analysis. The sixth sample, SL-00196, collected near the entrance to feeder No. 2 room, was overloaded by PCM analysis. Asbestos structures were detected in two of the six samples by TEM AHERA analysis, SL-00162 and SL-00163. SL-00162 was collected outside the lunch room in the main plant area, and SL-00163 was collected near the entrance to feeder No. 2 room.

Table 3-5
Stationary Air Sampling Results

Building	Location Description	Index ID	Sample Date	Sample Time (min)	PCM Lab Result (f/cc)	TEM AHERA Lab Result * (S/cc)
Central Maintenance	Center of machine shop	SL-00020	9/11/2002	480	<0.001	ND
Central Maintenance	Center of south end of building	SL-00021	9/11/2002	490	<0.001	ND
Central Maintenance	East side of center of building	SL-00022	9/11/2002	491	<0.001	ND
Central Maintenance	Center of north end of building	SL-00023	9/11/2002	479	<0.001	ND
Central Maintenance	Center of north end of building	SL-00024	9/11/2002	479	<0.001	ND
Plywood Plant	Green chain, exterior wall opposite supervisor's office	SL-00079	9/13/2002	184	0.01	ND
Plywood Plant	Plugger Alley, next to Plugger No. 9	SL-00081	9/13/2002	168	0.009	ND
Plywood Plant	Dryers, at post a feed end, near control panel	SL-00082	9/13/2002	135	0.005	ND
Plywood Plant	Dryers, at post a feed end, near control panel	SL-00090	9/13/2002	93	0.006	ND
Plywood Plant	Green chain, exterior wall opposite supervisor's office	SL-00091	9/13/2002	110	0.006	ND
Plywood Plant	Plugger Alley, next to Plugger No. 9	SL-00092	9/13/2002	72	0.021	ND
Plywood Plant	Dryers, at post a feed end, near control panel	SL-00094	9/13/2002	108	0.008	ND
Plywood Plant	Plugger Alley, next to Plugger No. 9	SL-00096	9/13/2002	137	0.005	ND
Plywood Plant	Green chain, exterior wall opposite supervisor's office	SL-00102	9/13/2002	87	0.014	ND
Plywood Plant	Dryers, at post a feed end, near control panel	SL-00106	9/13/2002	247	0.011	ND
Plywood Plant	Green chain, exterior wall opposite supervisor's office	SL-00107	9/13/2002	248	0.026	ND
Plywood Plant	Plugger Alley, next to Plugger No. 9	SL-00111	9/13/2002	242	0.008	ND
Plywood Plant	Debarker cab	SL-00215	9/17/2002	436	0.077	ND
Plywood Plant	Spreaders, at post near pre-press	SL-00243	9/18/2002	190	0.018	ND
Plywood Plant	Spreaders, at post near pre-press	SL-00245	9/18/2002	247	0.041	ND
Employee Parking Lot	Southeast corner	SL-00127	9/14/2002	465	0.001	ND
Employee Parking Lot	Center of south side of lot	SL-00128	9/14/2002	465	0.001	ND
Employee Parking Lot	Northwest corner	SL-00129	9/14/2002	457	0.001	ND
Employee Parking Lot	In railroad tracks, north of roadway	SL-00130	9/14/2002	459	0.002	ND
Finger Joint	Outside lunch room, in main plant area	SL-00162	9/16/2002	267	0.002	0.004
Finger Joint	Near entrance to Feeder No. 2 room	SL-00163	9/16/2002	266	0.001	0.004
Finger Joint	Near former lunch room	SL-00164	9/16/2002	266	0.004	ND
Finger Joint	Outside lunch room, in main plant area	SL-00195	9/16/2002	221	0.002	ND
Finger Joint	Near entrance to Feeder No. 2 room	SL-00196	9/16/2002	219	Overload	ND
Finger Joint	Near former lunch room	SL-00197	9/16/2002	218	0.005	ND
Log Yard	Outside log yard log truck scale shed	SL-00167	9/16/2002	233	0.001	ND
Log Yard	Outside log yard storage shed	SL-00168	9/16/2002	406	0.001	ND
Log Yard	At trailer crane by fire pond	SL-00181	9/16/2002	209	0.002	ND
Log Yard	Along service road, near head gate	SL-00182	9/16/2002	150	<0.002	ND
Log Yard	Outside log yard log truck scale shed	SL-00203	9/16/2002	188	0.002	ND
Log Yard	At trailer crane by fire pond	SL-00204	9/16/2002	193	0.002	ND
Log Yard	Along service road, near head gate	SL-00244	9/18/2002	424	0.001	ND
Central Maintenance	Center of north end of building	SL-00213	9/17/2002	218	<0.001	ND
Central Maintenance	Center of north end of building	SL-00214	9/17/2002	218	<0.001	ND
Central Maintenance	Center of north end of building	SL-00222	9/17/2002	293	0.001	ND
Central Maintenance	Center of north end of building	SL-00223	9/17/2002	293	0.001	0.003

* ND - Indicates no Libby Amphibole structures detected by TEM AHERA analysis.
AHERA - Asbestos Hazardous Emergency Response Act
f/cc - Fibers per cubic centimeter
min - Minutes
PCM - Phase contrast microscopy
S/cc - Structures per cubic centimeter
TEM - Transmission electron microscopy

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Of the four samples collected in the employee parking lot, fibers were not detected at levels at or above 0.01 f/cc by PCM analysis. Asbestos structures were not detected in any of the four samples by TEM AHERA analysis.

Of the seven samples collected in the log yard, fibers were not detected at levels at or above 0.01 f/cc by PCM analysis. Asbestos structures were not detected on any of the seven samples by TEM AHERA analysis.

Section 4
Microvacuum Dust Sampling

A total of 36 microvacuum dust samples were collected from buildings at Stimson. Stimson employees identified buildings as containing vermiculite or not containing vermiculite. One microvacuum dust sample was collected from each building not known to contain vermiculite. Up to five microvacuum dust samples were collected from each building known to contain vermiculite. All samples were composite samples consisting of two to three 100 centimeters squared (cm^2) sub-samples per cassette. All locations were approved by EPA prior to sampling.

4.1 Sample Locations

Microvacuum dust samples were collected from several buildings at Stimson. An overview of the entire property is shown on Figures 1-2 and 1-3.

Buildings that contain identified vermiculite in which five microvacuum dust samples were collected were:

- Central Maintenance Facility (BD-002098)
  - Machine shop
  - South end of central maintenance building
  - Center of central maintenance building
  - Northern end of central maintenance building
  - Supervisor's office and break room

- Plywood Plant (BD-002099)
  - Break room and office at finish end
  - Plugger area
  - Spreaders and finish end
  - Green chain
  - Dryer area

Buildings that do not contain vermiculite in which two microvacuum dust samples were collected:

- Finger joint building (BD-002097)
  - Former lunchroom (now parts storage)
  - Doorways & entrances

- Truck barn (BD-002110)
  - North side
  - South end of building

- Stimson office (BD-002269)

    - First floor
    - Second floor

- Buildings that do not contain vermiculite in which one microvacuum dust sample was collected:

    - Log yard break building (BD-002100)
    - Log yard storage building (BD-002101)
    - Log yard oil storage shed (BD-002102)
    - Log yard pump house (BD-002103)
    - Log yard truck scale shed (BD-002104)
    - Irrigation building (BD-002105)
    - Diesel fire pump house (BD-002106)
    - Double wide trailer (BD-002107)
    - Electric pump house (BD-002108)
    - Guard station at Libby Creek Bridge (BD-002109)
    - Steel storage (BD-002111)
    - Fire hall (BD-002112)
    - Wagner shed (BD-002260)
    - Electric motor shed (BD-002261)
    - Astrodome (BD-002262)
    - Pipe shed (BD-002263)
    - Storage & locomotive shed (BD-002264)
    - Power house office (BD-002265)
    - Power house (BD-002266)
    - Lumber kilns (BD-002267)
    - Shed 12 (BD-002268)

4.2 Sample Collection

All microvacuum dust samples collected between September 12 and September 18, 2002, were collected in accordance with the American Society for Testing Materials (ASTM) Standard D-5755-95, Standard Test Method for Microvacuum Sampling and Indirect Analysis Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (Appendix D). Up to three separate 100 cm^2 areas per cassette for a total of up to 300 cm^2 per cassette. Samples were collected in each 100 cm^2 area for 2 minutes or until all visible dust had been removed. Sampling was performed using 0.45 μm MCE filters. All sampling pumps were calibrated from 2.03 to 2.05 lpm prior to the sampling period and again at the end of the sampling period.

All field blanks analyzed during this investigation returned results of ND, or no structures detected at or above the detection limit for the analytical method. Field blank results are included in the Libby database printout presented in Appendix A.

Specific cassette lot blanks analyzed during this project included filters from Lot Number 410FKA.

All lot blanks analyzed during this investigation returned results of ND, or no fibers detected at or above the detection limit for the analytical method. Lot blank results are included in the Libby database printout presented in Appendix A.

4.3 Sample Analysis

Air samples were analyzed by EMSL Analytical, Inc. in Libby, Montana, and Westmont, New Jersey; Reservoirs Environmental Services in Denver, Colorado, and/or Hygeia in Sierra Madre, California. All samples were analyzed in accordance with the ISO 10312, Air Quality - *Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method, 1995.*

Samples were maintained under chain of custody procedures. After sample collection was completed, EPA custody labels were completed and affixed to each sampling cassette. Sample information was entered on chain of custody forms, and changes in sample custody noted on the form. Samples were relinquished to the CDM sample coordinator to be submitted for laboratory analysis. Any samples not immediately relinquished to the sample coordinator were secured in the sample storage cabinet. Samples were either hand-delivered to the EMSL onsite laboratory in Libby, Montana, or shipped via Federal Express to an offsite laboratory.

4.4 Summary of Results

LA structures were detected in two of the samples collected in the central maintenance building (BD-002098). Analysis identified 8823.1 LA structures with lengths between 0.5 μm and 5 μm in sample SL-00061, which was a composite of one locations in each of the Cummins engine room, Cat engine room, and large jack stand in the main work area. Analytical results indicate 882.31 LA structures with lengths between 0.5 μm and 5 μm were detected on SL-00064, which was a composite of three locations in the supervisor's office and break room (Table 4-1). Additionally, sample results for SL-00059 identified 4411.6 LA structures with lengths between 0.5 μm and 5 μm. Composites for this sample were collected in the machine shop.

LA structures were detected in one of the samples collected in the truck barn (BD-002110). Results identified 1971.3 LA structures with length between 0.5 μm and 5 μm and 985.7 LA structures with length between 5 μm and 10 μm were detected on SL-00225, which was a composite of three locations on the south side of the truck barn. No LA structures were detected in the samples collected on the north side of the truck barn (BD-002110, SL-00224) (Table 4-1).

LA structures were detected on both of the samples collected in the Stimson office building (BD-002269). Results for sample SL-00241 identified 262.84 LA structures with lengths between 0.5 μm and 5 μm, which was a composite of three locations on the first floor of the Stimson office building. Analysis also identified 131.42 LA

Table 4-1
Microvacuum Dust Sampling Results

Building	Location Description	Index ID	Subsample Locations (100 cm^2)	Sample Area (cm^2)	Lab Results* Excluded Structures Diameter > 0.5 µm	Libby Amphibole Length 0.5 µm to 5 µm	Libby Amphibole Length 5 µm to 10 µm	Libby Amphibole Length > 10 µm
Central Maintenance	Machine shop	SL-00059	Floor in front of sliding door to main area Floor in front of rear sliding door, opposite above Blade on large fan stored in rear corner	300	0	4411.6	0	0
Central Maintenance	South end of CM building	SL-00060	On workbench near machine shop door In front of third vehicle door from south end Center of fourth vehicle area from south end	300	0	0	0	0
Central Maintenance	Center of CM building	SL-00061	Workbench in rear of Cummins Engine Room Between 5th vehicle door from south & Cat engine rm. Top of large jack stand near door	300	0	8823.1	0	0
Central Maintenance	North end of CM building	SL-00062	On shelf in warehouse in NE corner of building On top shelf in NE corner of main work area Floor in center of north end of building	300	0	0	0	0
Central Maintenance	Supervisor's office and break room	SL-00064	Top of CB unit in supervisor's office Top of refrigerator in break room Top of soda machine in break room	300	0	882.3	0	0
Finger Joint	Former lunch room	SL-00065	Floor in front of front door Floor in front of rear door On shelf to left of front door	300	0	0	0	0
Finger Joint	Doorways & entrances	SL-00066	Floor at pedestrian entrance to break room Floor at west vehicle door Floor at entrance to wrap & stack area, from main area	300	0	0	0	0
Log Yard Break Building	NA	SL-00169	Floor at entrance Floor, doorway between rooms Top of microwave oven	300	0	0	0	0
Log Yard Storage Shed	NA	SL-00170	Floor at entrance Floor, center of room Top of workbench	300	0	0	0	0
Log Yard - Oil Storage	NA	SL-00171	Floor at entrance Floor, near end of tank On shelf	300	0	4411.6	0	0
Log Yard - Pump House	NA	SL-00172	Floor at entrance Floor next to engine base On engine base	300	0	0	0	0
Log Yard - Log truck scale shed	NA	SL-00173	First floor - floor at entrance First floor - desktop Second floor - doorjamb	300	0	0	0	0
Irrigation Building	NA	SL-00174	Floor at entrance Floor of doorway between rooms Floor near center of front (entrance) room	300	0	4411.6	0	0
	NA	SL-00175	Floor at entrance	300	0	4411.6	0	0

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Table 4-1
Microvacuum Dust Sampling Results

Building	Location Description	Index ID	Subsample Locations (100 cm^2)	Sample Area (cm^2)	Lab Results*: Excluded Structures Diameter > 0.5 µm	Lab Results*: Libby Amphibole Length 0.5 µm to 5 µm	Lab Results*: Libby Amphibole Length 5 µm to 10 µm	Lab Results*: Libby Amphibole Length > 10 µm
			Floor at entrance					
Diesel Fire Pump House			Top of cabinet / Floor near pump					
Double Wide Trailer	NA	SL-00176	Floor at entrance	300	0	0	0	0
			Floor in front of kitchen area cabinets					
			Floor at bathroom entrance					
Electric Pump House	NA	SL-00177	Floor at front entrance	300	0	4411.6	0	0
			Floor at entrance to extension room					
			Floor at rear entrance					
Guard Station at Libby Creek Bridge - North Gate	NA	SL-00178	Floor at entrance	300	0	0	0	0
			Floor at counter to left of door (when looking into booth)					
			Countertop to right of door					
Plywood Plant	Break rooms & offices at finish end	SL-00217	Break room, floor near door to plant	300	0	0	0	0
			Second floor - shift super office, floor near entrance					
			First floor - floor near entrance to plant, NW corner					
Plywood Plant	Plugger Area	SL-00218	Floor near Plugger No. 1	300	0	0	0	0
			Floor near Plugger No. 9, storage side					
			Floor near turntable					
Plywood Plant	Spreaders and finish end	SL-00219	Floor near spreaders	200	0	0	0	0
			Floor near spreaders					
Plywood Plant	Green chain	SL-00220	Floor along center of chain, plant side	300	0	0	0	0
			Floor outside lunch/smoking area					
			Floor near lathe					
Plywood Plant	Dryer area	SL-00221	Floor near entrance/break room/restrooms	300	0	0	0	0
			Floor near feeder for little dryer					
			Floor at offbearer end, under belt					
Truck Barn	North side	SL-00224	Floor near entrance	300	0	0	0	0
			Top of workbench/storage box					
			Floor towards rear of building					
Truck Barn	South side	SL-00225	Horizontal beam on dividing wall	200	0	1971.3	985.7	0
			Doorjamb floor					
Steel Storage	NA	SL-00226	Concrete floor	300	0	0	0	0
			Horizontal beam on dividing wall					
			Pipe stored in shed					
Fire Hall	NA	SL-00227	Floor at vehicle entrance	300	0	0	0	0
			Top of workbench					
			Third step up on stairs to second floor					
Wagner Shed	NA	SL-00228	Horizontal beam on side wall	200	0	394.3	0	0

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Table 4-1
Microvacuum Dust Sampling Results

Building	Location Description	Index ID	Subsample Locations (100 cm^2)	Sample Area (cm^2)	Lab Results* Excluded Structures Diameter > 0.5 μm	Lab Results* Libby Amphibole Length 0.5 μm to 5 μm	Lab Results* Libby Amphibole Length 5 μm to 10 μm	Lab Results* Libby Amphibole Length > 10 μm
			Shelf on rear wall					
Electric Motor Shed	NA	SL-00229	Floor at overhead door entrance On storage shelf Second floor at entrance to storage area	300	0	328.6	0	0
Astrodome	NA	SL-00230	Horizontal beam on long wall Floor near exposed corner Horizontal base beam on short wall	300	0	0	0	0
Pipe Shed	NA	SL-00231	Floor in front of door Top of workbench Top of storage shelf	300	0	0	0	0
Storage & Locomotive Shed	NA	SL-00232	Floor at center doorway Top of storage bin Between train rail tracks	300	0	375.5	375.5	0
Power House Office	NA	SL-00237	Floor in front of door Top of refrigerator	200	0	0	0	0
Power House	NA	SL-00238	Floor in front of door near office Floor in front of door near diesel tanks Horizontal beam in garage	300	0	0	0	0
Lumber Kilns	NA	SL-00239	Floor of infeed at first bay Floor in center of bay No. 15 Floor in center of tunnel of north side	300	0	0	0	0
Shed 12	NA	SL-00240	Floor at north entrance Horizontal beam on wall Floor at top of ramp to FJ building	300	0	0	0	0
Stimson Office Building	First Floor	SL-00241	Floor at back entrance Floor mat at front entrance Top of stairs to conference room	300	0	262.8	0	0
Stimson Office Building	Second Floor	SL-00242	Floor at back entrance Floor in front of men's room Top of refrigerator	300	0	131.4	0	0
Nursery Shed	Concrete Floor	1-06850	Center of main section West end of main West room, center of floor	300	0	0	0	0
Nursery Shed	Standing wood, debris removed from walls	1-06857	Top of wood piles at east end of main Top of wood pile at west end of main South wall, west room on ground level, horizontal beam	300	0	5853.1	1170.6	0

* Laboratory also reports chrysotile and other amphiboles.
See Appendix D for complete results.

NA - Not available
cm^2 - Square centimeters
μm - Micrometer

structures with lengths between 0.5 μm and 5 μm in sample SL-00242, which was a composite of three locations on the second floor of the Stimson office building (BD-002269).

Analytical results presented 4411.6 LA structures with lengths between 0.5 μm and 5 μm on four samples:

- SL-00171, a composite of three locations in the log yard oil storage building (BD-002102)
- SL-00174, a composite of three locations on the irrigation building floor (BD-002105)
- SL-00175, a composite of three locations in the diesel fire pump house (BD-002106)
- SL-00177, a composite of three locations on the electric pump house floor (BD-002108)

Analysis of sample SL-00228 identified 394.26 LA structures with lengths between 0.5 μm and 5 μm, which was a composite of two locations in the Wagner shed (BD-002260).

Analysis identified 328.55 LA structures with lengths between 0.5 μm and 5 μm in sample SL-00229, which was a composite of three locations in the electric motor shed (BD-002261).

Analytical results of sample SL-00232 identified 375.5 LA structures with lengths between 0.5 μm and 5 μm and 375.5 LA structures with length between 5 μm and 10 μm, which was a composite of three locations in the storage and locomotive shed (BD-002264).

LA structures were not detected on samples collected in the following buildings:

- Plywood plant (BD-002099)
- Finger joint building (BD-002097)
- Log yard break building (BD-002100)
- Log yard storage building (BD-002101)
- Log yard pump house (BD-002103)
- Log yard truck scale shed (BD-002104)
- Double wide trailer (BD-002107)
- Guard station at Libby Creek bridge (BD-002109)
- Steel storage (BD-002111)
- Fire hall (BD-002112)
- Astrodome (BD-002262)
- Pipe shed (BD-002263)
- Power house office (BD-002265)
- Power house (BD-002266)
- Lumber kilns (BD-002267)
- Shed 12 (BD-002268)

Section 5
Quality Assurance

The field quality assurance program was designed in accordance with CDM's RAC VIII *Quality Management Plan,* Revision 1 (CDM 2002d).

5.1 Adherence to the Sampling and Analysis Plan

All sampling was completed in accordance with the *Property Specific Sampling and Analysis Plan (SAP), Air and Dust Sampling for Stimson Lumber Company, Libby Asbestos Project, Libby, Montana* (CDM 2002a). Deviations from the SAP are addressed in Section 5.2.

5.2 Deviations

All of the personal samples were collected in accordance with the SAP. Deviations were made while collecting ambient air samples and dust samples, as discussed in Section 5.2.1.

5.2.1 Deviations During Ambient Air Sample Collection

According to the SAP, approximately four ambient samples were to be collected at each specific sampling location. This number was changed to collect samples more representative of normal working conditions in each location. Additional samples were collected in the larger buildings. Five ambient samples were collected in the central maintenance building and the plywood plant, and three samples in the FJ building.

Normal work activities at Stimson generated significant amounts of airborne particulates. In an effort to collect more representative ambient air samples and prevent filter overload, less than the standard volume of air (4,000 liters) for 33 ambient air samples was collected. Also, less than 4,000 liters was collected during sample recollection. Samples were recollected when the laboratory indicated that previous samples collected in that location were overloaded.

On September 11, 2002, ambient samples were collected in the central maintenance building. Greater than 4,000 liters of air was collected for these samples, but there was concern about sample overload since the filters appeared to be more than 30 percent loaded by visual inspection. On September 16, 2002, samples were recollected in the same location. Less than 4,000 liters of air was collected for the resampling to prevent sample overload.

On September 13, 2002, ambient samples were collected in the plywood plant. On that date, there were high levels of visible airborne dust throughout the plant. Sample cassettes were changed out at less than 4,000 liters to prevent filter overloading. Plant employees and Ms. Bovee later explained that the bag house was not functioning properly on that date. The bag house collects airborne particulates from the plant. On September 13, 2002, the "bags" were overfull, which resulted in higher than normal levels of airborne dust in the plant. According to Ms. Bovee, the bags were changed out on September 15, 2002. Samples were recollected near the spreaders on September 18, 2002. Less than 4,000 liters of air was collected to avoid filter overloading.

The debarker is located outside the plant building but generates a significant amount of sawdust during normal operation. On September 13, 2002, samples were collected outside the debarker's operator cab in the very dusty environment of the debarker. These cassettes were changed out at less than 4,000 liters. A sample was recollected inside the debarker cab on September 17, 2002. Less than 4,000 liters of air was collected to avoid filter overloading. Ambient air sampling cassettes collected in the FJ building and log yard were changed out at less than 4,000 liters to avoid filter overloading.

According to the SAP, replicate ambient air samples were to be collected at a rate of one per sampling location. In two locations (log yard and parking lot), insufficient electrical supply made replicate sampling infeasible. In addition, replicate samples collected in the plywood plant were among those that were overloaded and unreadable. As a result, three readable replicate samples were analyzed during this project.

5.2.2 Deviations During Dust Sample Collection

According to the SAP, one dust sample was to be collected in buildings that do not contain identified vermiculite. In three such buildings, two dust samples were collected. Due to the large size of these buildings, two samples would better characterize the interior of the building space.

5.3 Corrective Actions

In an effort to prevent ambient air sample overload and prevent indirect sample preparation, samples were recollected using reduced total sample volumes and/or collected with multiple representative samples.

5.4 Discussion of Quality Control Results

Laboratory analysis of the lot blanks indicated asbestos fiber counts were below the detection limit of the analytical method. Laboratory analysis of the field blanks indicated asbestos fiber counts were below the detection limit of the analytical method. The QC results indicate that the sampling cassettes were not contaminated with asbestos when received from the supplier and that cassettes were not contaminated when they were handled in the field.

Laboratory analysis of the replicate ambient air samples indicated that the replicate sample results were within ±10 percent of the adjacent ambient air sample. The results of the replicate samples indicate that sample variability was within an acceptable range.

Section 6
References

American Society for Testing and Materials (ASTM). 1995. Standard D-5755-95, Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations.

CDM Federal Programs Corporation (CDM). 2002a. *Property Specific Sampling and Analysis Plan (SAP), Air and Dust Sampling for Stimson Lumber Company, Libby Asbestos Project, Libby, Montana.*

_________. 2002b. Final Sampling and Analysis Plan (SAP) for the Remedial Investigation (RI) Contaminant Screening Study (CSS), Libby Asbestos Site, OU4. Libby, Montana. April.

_________. 2002c. Final SAP Addendum for the Stimson Lumber Company Area, Libby Asbestos Site, OU4. Libby, Montana.

_________. 2002d. *Quality Management Plan,* Revision 1.

International Organization of Standards (ISO) 10312. 1995. Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method.

National Institute of Occupational Safety and Health (NIOSH). 1995. Method 7400. Asbestos and other Fibers by Phase Contrast Microscopy (PCM).

U. S. Environmental Protection Agency (EPA). 2002. Action Memorandum Amendment for the Time-Critical Removal Action at the Libby Asbestos Site, Libby, Lincoln County, Montana. May.

TARGET SHEET

EPA REGION VIII

SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 2009598

SITE NAME: LIBBY ASBESTOS

DOCUMENT DATE: 12/01/2002

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☑ TYPES OF DOCUMENTS NOT TO BE SCANNED
  (Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

APPENDIX A Analytical Data Sheets

Contact the Superfund Records Center to view available document.
(303) 312-6473

Appendix B

EPA SOP 2015

ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 µm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and medical surveillance[5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection, is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.

- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.

- There is increased opportunity for fiber loss or introduction of extraneous contamination.

- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m^3).

	Concentration	Flow Rate
• Low RAM readings:	<6.0 mg/m^3	11-15. L/min
• Medium RAM readings:	>6.0 mg/m$_3$	7.5 L/min
• High RAM readings:	>10. mg/m^3	2.5 L/min

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump.Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling *pump, rotameter, and electronic calibrator.*

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Upwind/Background[1]	Collect a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

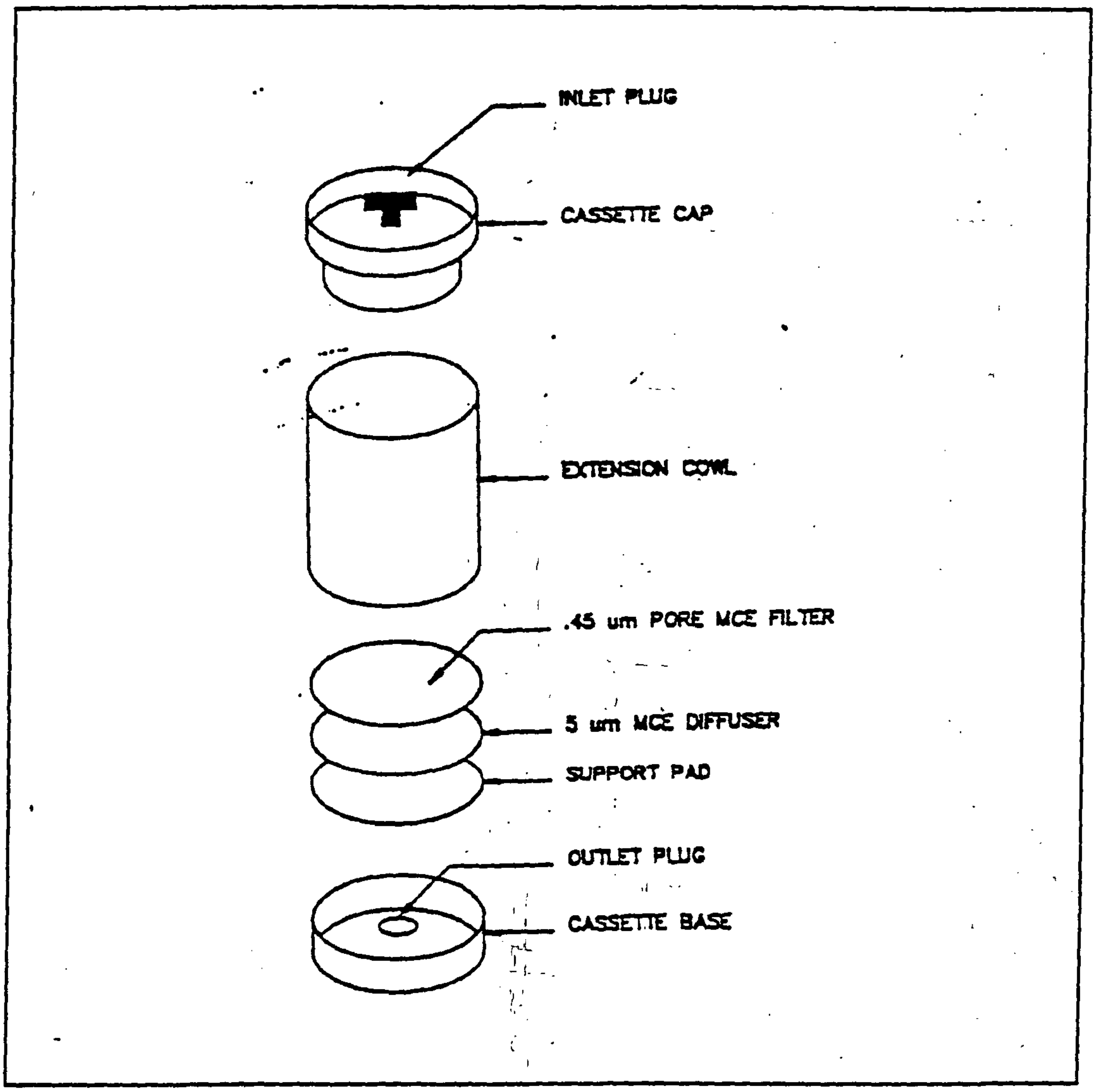
Tables

TABLE 2 SAMPLE STATIONS FOR INDOOR SAMPLING		
Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

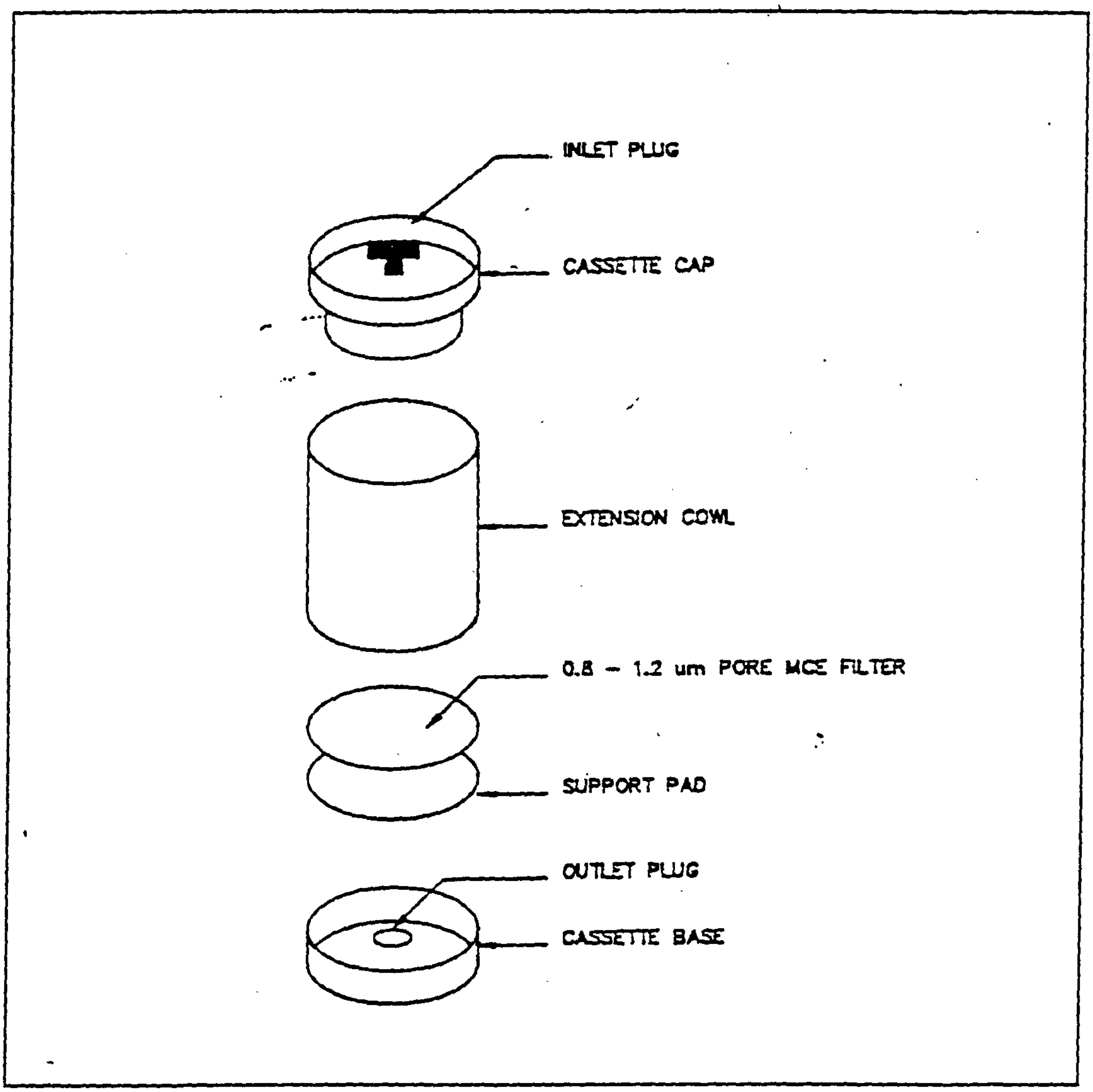
FIGURE 1. Transmission Electron Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

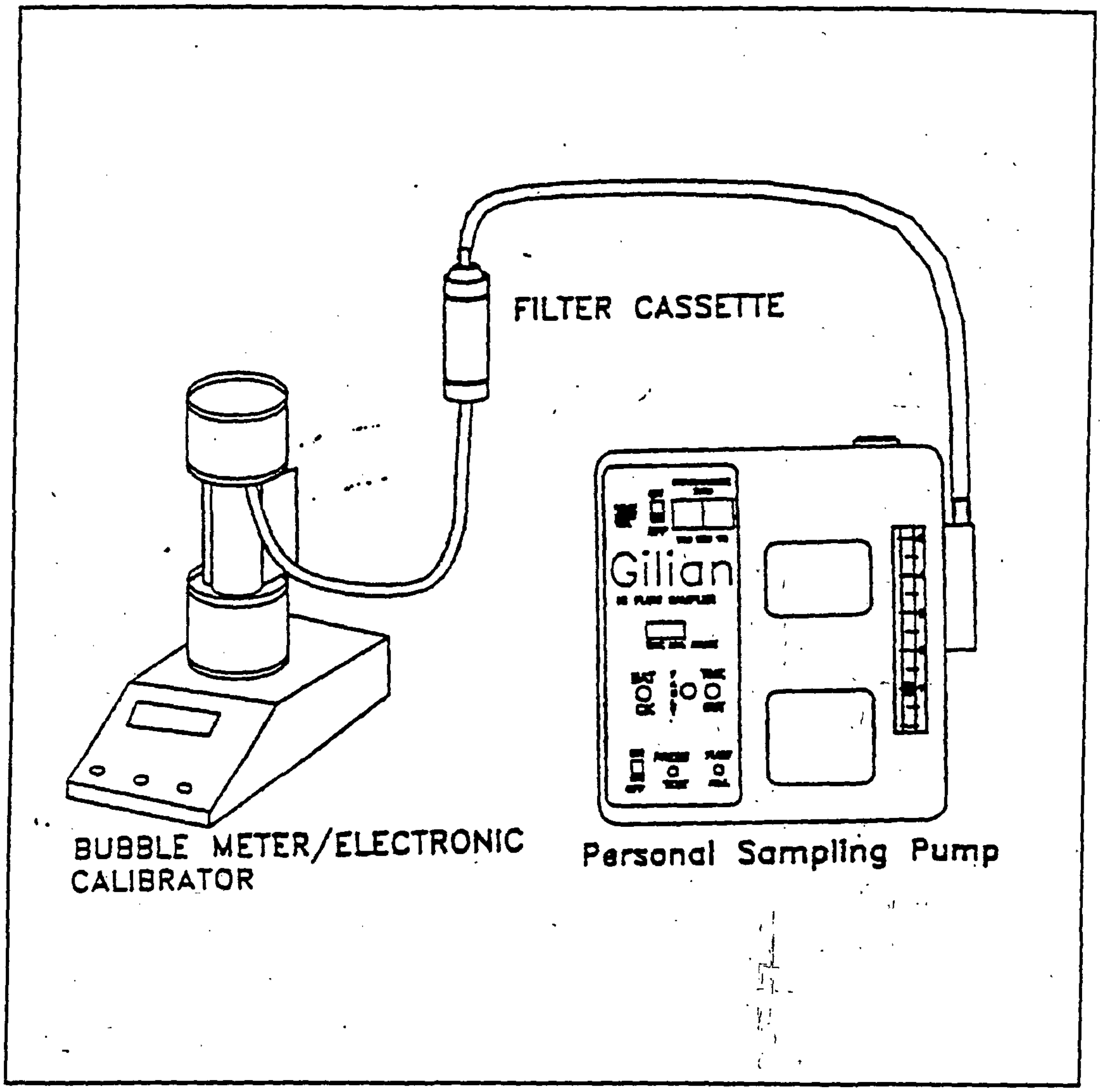
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

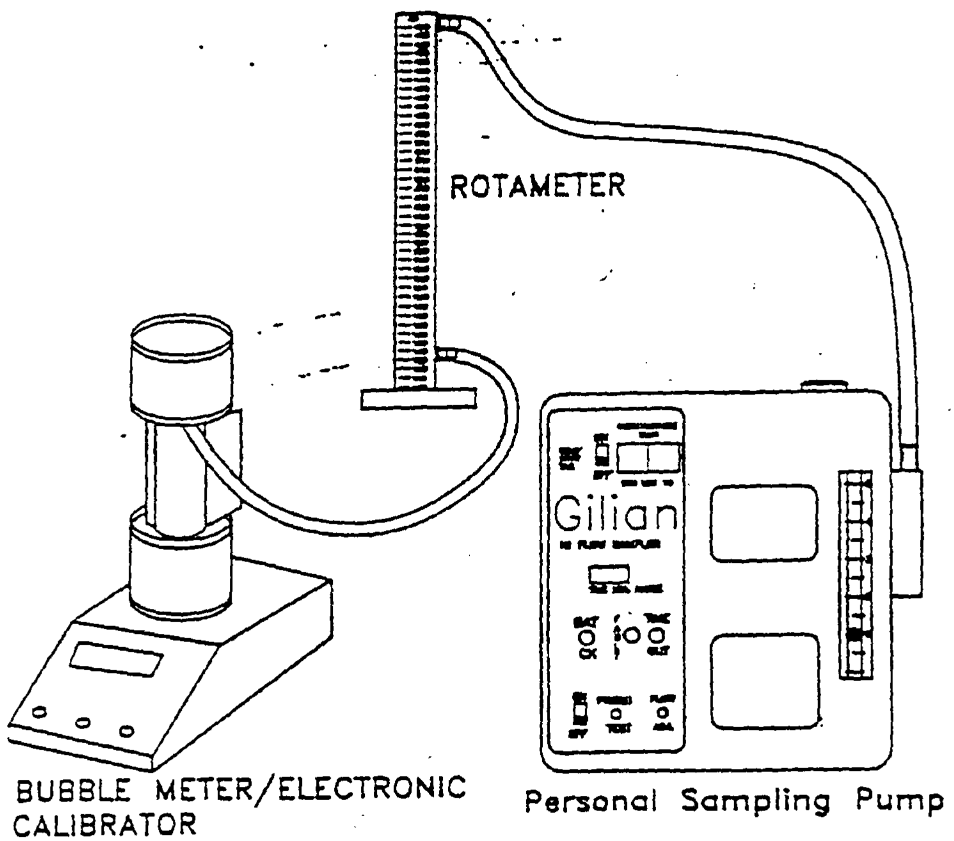
FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



APPENDIX B (Cont'd)

Figures

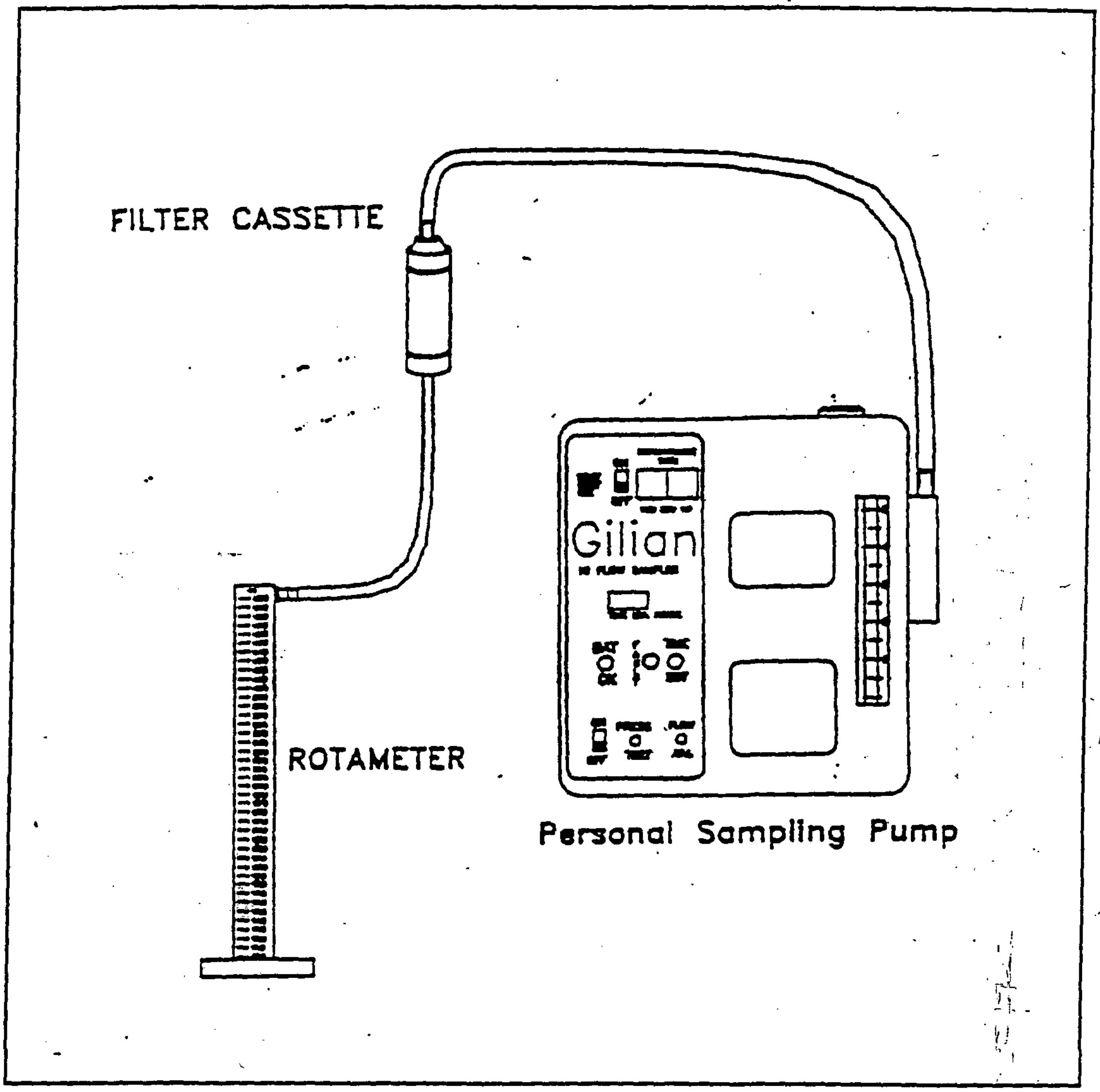
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

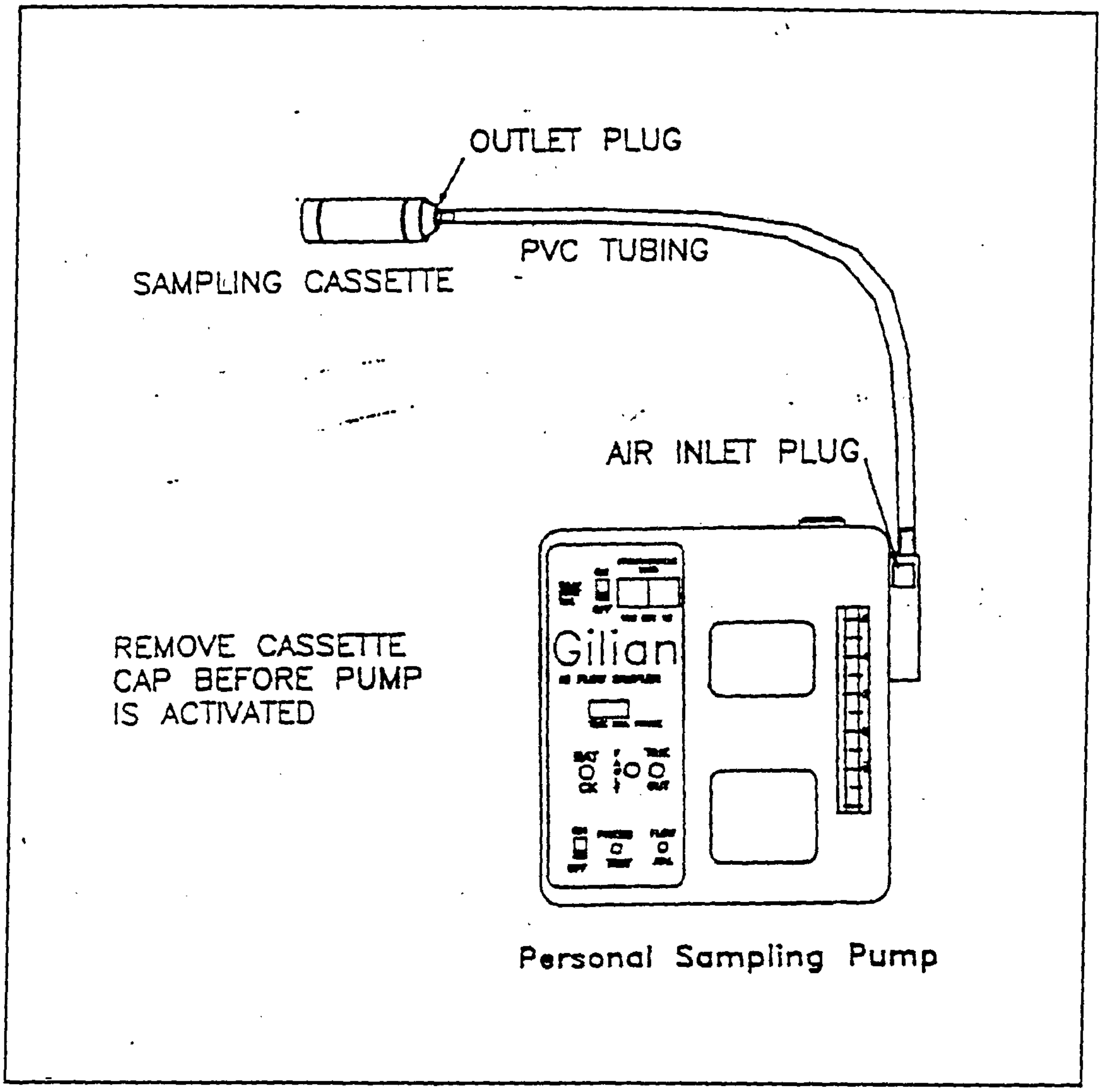
Figures

FIGURE 5. Calibrating a Sampling Pump with a Rotameter



Figures

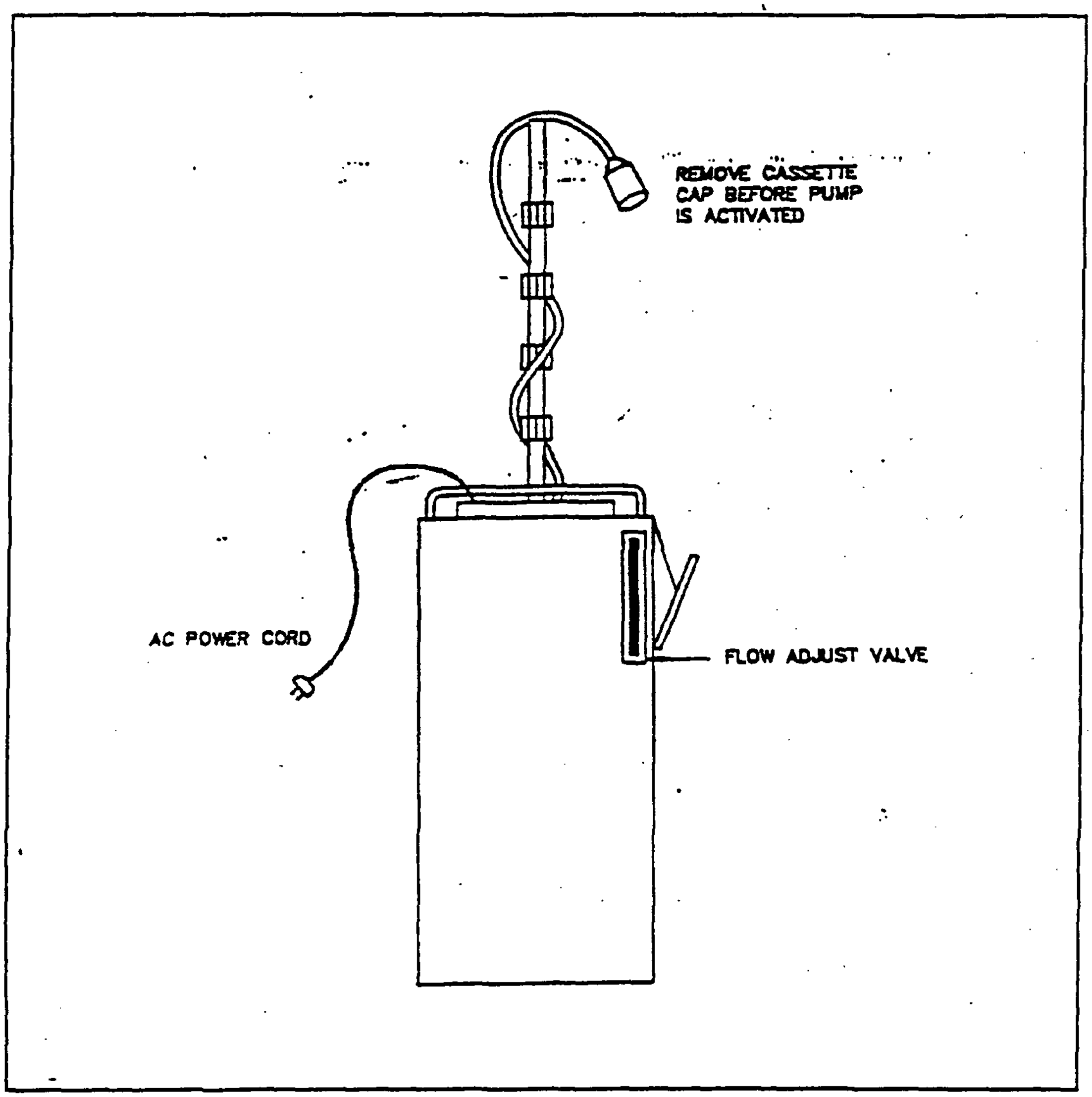
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Appendix C

Employee Orientation Form

Personal Air Sampling

There are a few important points to remember when wearing a personal sampling pump:

1) The cassette needs to be located in your breathing zone (near mouth & nose) throughout your workday. It should be attached to your lapel, or in a comparable location. Please do not move (or remove) the cassette without the assistance of the technician. The technician will pause the pump prior to and during breaks and lunch.

2) Please advise the technician as soon as possible if there are ANY irregularities with the pump or cassette. Examples include:

- Cassette falls off or is damaged
- Pump falls off, stops running, or is damaged
- Hose is cut or damaged

This will enable the technician to adjust, restart, or replace your pump as necessary to provide an accurate, valid representation of the air sampled.

3) Please advise the technician of any non-routine occurrences during the workday. This would include any accidents, unusual outages, variations in standard procedures, assignment to a different task, or any other changes to your normal daily routine. You will not be penalized for any of these changes – it ensures that we collect valid air samples.

Please sign to acknowledge review of these items.

Signature ________________________________

Printed Name________________________________

Last four digits of SSN (for sample tracking purposes) ____________

Task ________________________________

Date ________________________________

Appendix D

American Society for Testing and Materials

D-5755-95

Designation: D 5755 – 95

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)[3]
D 3195 Practice for Rotameter Calibration[3]
D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-22[3]
D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1),[4] (2) and (3).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

DISCUSSION—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into

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[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved August 15, 1995. Published October 1995.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[5] The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

smaller components without losing its fibrous properties or appearance.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).

3.2.6 *fiber*—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of nonfibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition, settleable particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[4]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8 µm or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon[7] Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 Record all calibration information (5).

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

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[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC),

[7] Tygon is a registered trademark of the DuPont Co.

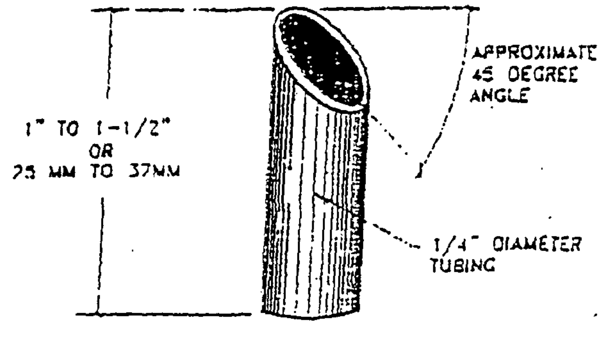

FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 μm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 μm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (*1*) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (*2*) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method:*

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method:*

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer:*

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

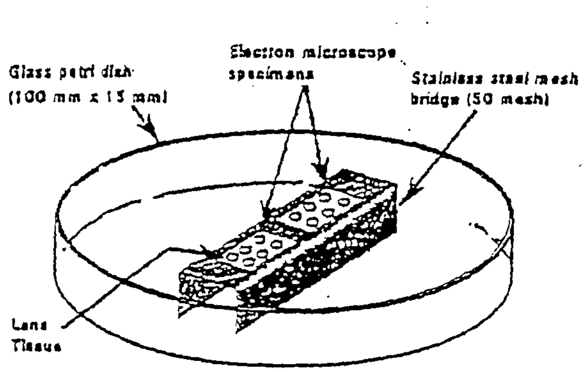

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ¼ mark) DMF Jaffe washer for 60 min. Add enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2: Caution—Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing*

Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules:*

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity*—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 *Limit of Detection*—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analytical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the laboratory report.

16.10 *Stopping Rules:*

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 asbestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm^2.

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ mL \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \qquad (1)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM:*

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(*a*) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(*b*) Frequency of calibration will depend on the service history of the particular microscope.

(*c*) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(*d*) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(*e*) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(*a*) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(*b*) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA:*

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

... versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \qquad (2)$$

where:

4.185 = Joules/cal,

R = energy deposition, watts/mL,

θ_1 = temperature rise with the ultrasonic bath not operating, °C,

θ_2 = temperature rise with the ultrasonic bath operating, °C,

t = time in seconds, 60 s (20.5.3 and 20.5.5),

σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and

ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client: ____________________

Sample ID: ____________________

Job Number: ____________________

Data Sample Analyzed: ___ - ___ - ___

Number of Openings/Grids Counted: ___ | ___

Grid Accepted, 600X: Yes No

Percent Loading: ______ %

Grid Box #1: ____________________

Accelerating Voltage: ____________________

Indicated Mag: ______ KX

Screen Mag: ______ KX

Microscope: 1 2 3 4 5

Filter Type: ____________________

Filter Size: ____________________

Filter Pore Size (μm): ____________________

Grid Opening: 1) ______ μm x ______ μm

2) ______ μm x ______ μm

Analyst: ____________________

Reviewer: ____________________

Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	______
Number of Grid Openings Counted:	(GO)	______
Avderage Grid Opening Area in mm^2:	(GOA)	______
Volume of sample Filtered in ml:	(V)	______
Surface area Sampled in cm^2:	(SPL)	______
Number of Asbestos Structures Counted:*	(#STR)	______

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFA \times 100 \times \#STR}{GO \times GOA \times V \times SPL} = \text{(Asbestos Structures per cm}^2\text{)}$$

Results for Total Asbestos Structures: ____________________
(Structures per cm^2)

Results for Structures ≥ microns: ____________________
(Structures per cm^2)

FIG. X1.1 Dust Sample Analysis Work Sheet

Job Number: ______________________

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

Note: *Keys to Abbreviations Used in Figure:*

Type:

- C = Chrysotile
- AM = Amosite
- CR = Crocidolite
- AC = Actinolite
- TR = Tremolite
- AN = Anthophyllite
- N = Non Asbestos

Structure:

- F = Fiber
- B = Bundle
- C = Cluster
- M = Matrix

Others:

- NSD = No Structures Detected
- Morph = Morphology
- SAED = Selected Area Electron Diffraction
- EDS = Energy Dispersive X-Ray Spectroscopy
- ER = Inter-Row Spacing
- NP = No Pattern

FIG. X1.2 TEM Count Sheet

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